# **DECORATIVE CONTAINER**

# Field of the Invention

The present invention is directed to a decorative container, more particularly a decorative container including a hollow region where objects can be displayed.

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# **Background of the Invention**

Decorative containers are known in the prior art that allow sheets of material or objects to be stored within the walls of the container. For example, some beverage mugs provide space for a sheet of material, such as a photo, in the wall of the mug. Some of these decorative mugs may allow for insertion of liquid or fine particles within the space provided for decoration.

A transparent bowl is known that provides a hollow region between an inner and outer bowl that is purchased with decorative objects already present in the hollow region. However, it is not possible for the purchaser of this bowl of the prior art to access the hollow region to change the decorative objects.

Transparent storage systems for items such as fishing lures are also known, such as the compartmentalized container of U.S. Patent No. 3,378,134. The compartments can be rotated to bring them in line with access openings on the outer container wall. The access openings may be covered by a hinged access door that is readily visible on the container wall. The access door provides access to only one compartment at a time.

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A decorative container providing a hollow region for displaying three-dimensional decorative objects is described in U.S. Patent Application Serial No. 09/586,153, filed June 2, 2000. This container provides dividers within the hollow region for separating the decorative objects in a number of internal cavities and a removable access opening for accessing the hollow region.

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There is a need for additional arrangements for holding three-dimensional decorative objects in a decorative container or decorative container system. Containers with different configurations are needed where decorative objects in a hollow region of the container can be changed by the user.

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# Description

# FIELD OF THE INVENTION

This invention generally relates to aqueous dispersion slurry of inorganic particles and production methods thereof. Aqueous dispersion slurry produced by production methods according to this invention are free from such problems as increase in viscosity, gelation and separation/sedimentation during storage, i.e., highly stable. For this reason, such aqueous colloidal dispersion slurry may be suitably used, for example, as raw materials for cosmetics, paint, coating materials and polishing slurry for semiconductor waters.

# BACKGROUND OF THE INVENTION

As raw materials for cosmetics, paint, coating materials and polishing slurry for semiconductor wafers, raw materials of high purity with a minimal concentration of impurities, such as inorganic particles synthesized by a vapor phase method, such as fumigation, (hereinafter referred to as "vapor phased inorganic particles") have been used. However, as the vapor phased inorganic particles are intensive in secondary coagulation, when the vapor phased inorganic particles are dispersed in the water, coagula of such vapor phased inorganic particles should be destroyed and fused in the water. If the coagula are not completely destroyed or fused, prob-Iems, such as increase in viscosity of the aqueous dispersion slurry as the time passes, gelation and consequent loss of fluidity, and sedimentation and separation of the coagula. As a result, the aqueous dispersion slurry of inorganic particles can no longer be applied to those uses as described above.

As a method of dispersing the vapor phased inorganic particles in an aqueous medium, a method using dispersion equipment of high-speed mixing type (e.g., whirling blender, and high shearing mixer) as disclosed in USP5116535 (Japanese Unexamined Patent Publication No. 3-50112) has been known. Also, a method using equipment combining a powder introduction type mixer/disperser (e.g., jet stream mixer) with a toothed colloid mill, a dissolver or a skim mixer (Nippon Aerosil Co., Ltd.; Catalog No. 19: How to Handle Aerosil, p. 38) has also been known. However, both of these methods are disadvantageous in that a long-time processing is required and that vapor phased inorganic particles can not be completely destroyed or fused.

As regards the method of dispersing pulverized silica or fumed silica in an aqueous medium, a method of pulverizing and dispersing the vapor phased inorganic particles using a high-pressure homogenizer to 100 nm or less in average secondary particle diameter has been disclosed in the Japanese Unexamined Patent Publication No. 9-142827 (opened on June 3, 1997) and in the Japanese Unexamined Patent Publication No. 9-193004 (opened on July 29, 1997), which were opened

after the date of application of the Japanese Patent Application No. 9-132881 over which the present invention claims priority. However, these publications contain no description of the dispersion of inorganic particles other than the pulverized silica and the fumed silica. Under these circumstances, development of a method of producing the aqueous dispersion slurry of various inorganic particles has been wanted.

# SUMMARY OF THE INVENTION

It is a primary object of the present invention to provide aqueous dispersion slurry of inorganic particles of 0.01 to 2 µm in average diameter which are highly stable with no increase in viscosity, no gelation or no sedimentation even if stored for a long time, and production methods of such aqueous dispersion slurry of inorganic particles. As such production methods, the following 2 production methods, (A) and (B), are available:

# 1. PRODUCTION METHOD (A)

The production method (A) is characterized by performing a preliminary dispersion process in which inorganic particles are added to and dispersed in an aqueous medium and a main dispersion process in which the dispersed slurry out of the preliminary dispersion process are collided against each other or against walls of a disperser under a pressure of 100 to 3,000 kg/cm<sup>2</sup>.

In the production method (A), preferably, the dispersion slurry out of the main dispersion process should be quickly placed under a normal pressure, and more preferably, the dispersion slurry should be quickly placed under a normal pressure by flowing the same dispersion slurry at a high speed.

In the preliminary dispersion process, preferably the inorganic particles should be 3 to 70wt% in concentration in which the inorganic particles are suitable for the production of aqueous dispersion slurry. More preferably, the concentration of the inorganic particles should be 4 to 60wt%. If the concentration of the inorganic particles in the preliminary process is less than 3wt%, the dispersion efficiency will drop to such an extent that the obtained aqueous dispersion slurry of inorganic particles may quickly become unstable. On the other hand, if the concentration of the inorganic particles in the preliminary process exceeds 70wt%, the inorganic particles may not be sufficiently wet due to too small water content, or the viscosity of dispersing liquid may excessively increase or the inorganic particles may be gelled and become less fluid.

The main dispersion process (i.e., collision process) can be performed by using a high-pressure homogenizer which allows the dispersion slurry out of the preliminary dispersion process to be guided through a plurality of pipes and then joined with each other. FIG. 1 (a) shows a concept of the high-pressure homoge-

nizer which guides the dispersion slurry out of the preliminary process through 2 pcs of pipes 51 and 52, collides the dispersion slurry out of the pipes 51 and 52 against each other under the foregoing pressure, and then feeds the dispersion slurry through a drain pipe 30 at a high speed, branches the dispersion slurry into 2 pcs of branch pipes 61 and 62, and then collects the dispersion slurry into a collection tank (not illustrated). FIG. 1 (b) shows an example in which the dispersed slurry out of the preliminary dispersion process are guided from a main pipe 50a into 2 pcs of branch pipes 51a and 52a branched from the main pipe 51a, collides the dispersion slurry out of the branch pipes 51a and 52a against each other, and then upwardly discharges the dispersed slurry.

In these examples, the aqueous dispersion slurry out of the preliminary dispersion process collide against each other. Here, the main dispersion process may be performed by having the dispersion slurry out the preliminary dispersion process collided against pipe walls instead of having the dispersion slurry out of the preliminary dispersion process collided against each other.

To quickly place the dispersion slurry after collision under a normal pressure, only what to do is to have the dispersion slurry flow at a fast speed from the collision position into a sufficiently large container.

# 2. PRODUCTION METHOD (B)

The production method (B) is a production method for the aqueous dispersion slurry of inorganic particles which is characterized by that inorganic particles are added to an aqueous medium within a kneading tank of a kneader in which a mixture of the inorganic particles and the aqueous medium is to be kneaded by rotation of mixing blades around respective subsidiary spindles and revolution of the subsidiary spindles around a spindle, and the inorganic particles are dispersed through such kneading at a solid concentration of 30 to 70wt%.

Here, the kneader in which mixing blades rotate around respective subsidiary spindles and the subsidiary spindles revolve around a spindle is generally called "a planetary kneader."

In the production method (B), the concentration of the inorganic particles in dispersion in an aqueous medium should be 30 to 70wt%, preferably be 35 to 60wt%, and more preferably be 40 to 50wt%. If the solid concentration of the inorganic particles is less than 30wt%, the dispersion efficiency will drop to such an extent that coagula may be left in the obtained aqueous dispersion slurry, and as a result, such coagula may sediment and separate during storage or gel due to increase in viscosity. On the other hand, if the solid concentration of the inorganic particles exceeds 70wt%, load on the disperser will become too large to maintain mixing in motion. If mixing is forced in such state, the inorganic particles may be excessively dispersed, and as a result, a large quantity of coarse particles of 10 µm

or more in particle diameter may be produced due to recoagulation.

In the production method (B), it is preferable that the inorganic particles should be dispersed in an aqueous medium while continuously and intermittently added. If the inorganic particles are added in a required quantity at a time in the beginning, there will be problems that it is difficult to uniformly disperse the inorganic particles and the mixer may be stopped by an excessively large load. As an ideal method of adding the inorganic particles, it is preferable that the inorganic particles should be quickly added until the solid concentration reaches approx. 20wt% and then continuously and intermittently added while monitoring current value of (load on) the kneader not to be too large. As a hopper for supplying the inorganic particles, a screw transfer type or the like may be named.

Preferably, the aqueous dispersion slurry out of the dispersion process in the production method (B) should be diluted after the kneading process. The extent of such dilution depends on the type of the dispersed inorganic particles or the solid concentration in the kneading process. Preferably, the solid concentration should be decreased by 5wt% or more down from the solid concentration in the kneading process. If the solid concentration in the kneading process is maintained, as this solid concentration is too high, the inorganic particles are not only difficult to be processed but also may quickly gel due to increase in viscosity during storage.

As a dilution method, additionally applying an aqueous medium directly into the kneader is preferable as this way of dilution makes it easy to remove the dispersion slurry from the kneader.

In the production method (B), it is possible to perform the dispersion processing by using another kneader or disperser after the kneading process to improve the uniformity. In this case, for example, corres type high-speed mixer/disperser, homo-mixer, highpressure homogenizer or bead mill can preferably be used for this purpose.

### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 (a) is a descriptive view showing a principle of a high-pressure homogenizer, while FIG. 1 (b) is a descriptive view showing a principle of a high-pressure homogenizer which is different from that of FIG. 1 (a); and

FIG. 2 is a schematic view showing a planetary kneader, wherein FIG. 2 (a) is a top view thereof, and FIG. 2 (b) is a side view thereof.

# **DETAILED DESCRIPTION OF THE INVENTION**

Now, the present invention will be described in detail.

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# 1. INORGANIC PARTICLES

As a production method of the inorganic particles used in production methods (A) and (B) according to the present invention, such as a vapor phase method, a wet method and a sol gel method may be named. As the vapor phase method, a fumed method (high-humidity flame hydrolysis method), and a Nanophase Technologies Corporation method (metal evaporation and oxidation method) may be named. Furthermore, a production method in which natural inorganic compounds are pulverized, refined and/or classified can be named. Preferably, the inorganic particles should be produced by the vapor phase method, and more preferably, by the fumed method. The inorganic particles produced by the vapor phase method are highly purified and therefore favorable. The inorganic particles produced by the fumed method are highly purified and also comparatively economical and therefore particularly favorable.

As the inorganic particles preferably used in production methods (A) or (B), metallic oxides may be exemplified, such as silicon oxide, aluminum oxide, titanic oxide, ziruconium oxide, antimony oxide, chromium oxide, germanium oxide, vanadium oxide, tungusten oxide, iron oxide, manganese oxide and cerium oxide. More preferable among these metallic oxides are silicon oxide, aluminum oxide, titanic oxide and cerium oxide. These metallic oxides may be used singly or as a mixture of two or more.

As the particularly favorable inorganic particles used in production methods (A), metallic oxides may be exemplified, such as aluminum oxide, titanic oxide, ziruconium oxide, antimony oxide, chromium oxide, germanium oxide, vanadium oxide, tungusten oxide, iron oxide, manganese oxide and cerium oxide. More preferable among these metallic oxides are aluminum oxide, titanic oxide and cerium oxide. These metallic oxides may be used singly or as a mixture of two or more.

Typically, the inorganic particles to be dispersed are in a form of powder existing as coagula (called "secondary particles") of small particles (called "primary particles"). The average diameter of these primary particles is normally 0.005 to 1  $\mu$ m.

# 2. DISPERSION PROCESS AND EQUIPMENT

Although there is no particular limitation to equipment to be used in the preliminary dispersion process of the production method (A), such equipment that can directly disperse the inorganic particles into an aqueous medium while sucking the inorganic particles is preferable to preliminarily disperse a large quantity of the inorganic particles. Among such equipment is a powder introducer/mixer/disper (e.g., Jet Stream Mixer "Jet Stream Mixer" is a tread mark) of Mitamura Riken Kogyo Inc.).

To disperse the inorganic particles at a high concentration in the preliminary dispersion process according to the production method (A), equipment having a kneader with planetary motion type mixing blades or a high-speed rotation dispers (blades) is preferable. More preferably, such equipment should have a combination of a kneader in which the planetary motion type mixing blades and high-speed rotation dispers. Among such equipment are Planetary Disper ("Planetary Disper" is a tread mark) of Asada Iron Works Co., Ltd. and T.K. Hibis Disper Mix" is a tread mark) of Tokushu Kika Kogyo Co., Ltd.

Furthermore, a bead mill may be used in the preliminary dispersion process of the production method (A). The diameter of the beads to be used should be 0.1 to 10 mm, and preferably be 0.2 to 5 mm. If the bead diameter is smaller than 0.1 mm, it will be difficult to separate beads from the aqueous dispersion slurry after production. If the bead diameter is larger than 10 mm, collision of the inorganic particles against the beads will be less frequent, and as a result, not only the dispersion efficiency will drop but also beads may be broken and broken pieces may mix into the aqueous dispersion slurry of inorganic particle.

Among preferable materials of beads are non-alkali glass, alumina, zircon, zirconia, titania and silicon nitride. Preferably, a rotor or vessel (inner cylinder) of the bead mill should be provided with a lining resin, such as polyurethane, to protect the aqueous dispersion slurry from metallic contamination, or a lining of ceramic, such as zirconia, to prevent metallic contamination and increase wear resistance.

The preliminary dispersion process of the production method (A) may be performed for plural times on single equipment or for a single time on plural, different equipment.

The main dispersion process of the production method (A) is performed on equipment, such as a highpressure homogenizer which pressurizes the dispersion slurry out of the preliminary dispersion process to 100 to 3,000 kg/cm<sup>2</sup>. Among the high-pressure homogenizers available in the market and applicable to the main dispersion process are Manton Gaulin Homogenizer ("Manton Gaulin Homogenizer" is a tread mark) of Doei Shoji Co., Ltd., Bertoli Homogenizer ("Bertoli Homogenizer" is a tread mark) of Japan Machinery Company, Microfluidizer ("Microfluidizer" is a tread mark) of Mizuho industrial Co., Ltd., Nanomizer ("Nanomizer" is a tread mark) of Tsukishima Kikai Co., Ltd., Genus PY ("Genus PY" is a tread mark) of Genus Co., Ltd., System Organizer ("System Organizer" is a tread mark) of Nippon BEE Co., Ltd., and Ultimaizer ("Ultimaizer" is a tread mark) of Ito Industrial Machine Co., Ltd. Preferable equipment among those named are those which use sintered diamonds or single crystal diamonds for the liquid contacting portions of the dispersion processing unit. This is because if the liquid contacting portions of the dispersion processing equipment is made of ceramic or metal, the orifice will be widened due to wear, the passing liquid pressure will drop, and as a



result, dispersion may become incomplete. Furthermore, foreign matters produced by wear will mix into the dispersion slurry, and the purity of the dispersion slurry will drop to such an extent that the purity can not be practically acceptable.

The pressure to be applied by the high-pressure homogenizer or the like should be in a range of 100 to 3,000 kg/cm², and preferably be in a range of 200 to 2,000 kg/cm². If such pressure is lower than 100 kg/cm², dispersion will be incomplete. If such pressure is higher than 3,000 kg/cm², disadvantageously larger-sized equipment will be required and this will raise the processing cost. In the main dispersion process, the processing on the high-pressure homogenizer may be performed for plural times.

In the production method (B), a kneader which performs a planetary motion, i.e., mixing blades rotate around respective subsidiary spindles and at the same time the subsidiary spindles revolve around a spindle can be used. Such kneader should preferably be equipped with high-speed rotation dispers (blades) as well.

Here, the equipment in which the mixing blades perform the planetary motion will be described. FIG. 2 schematically shows a planetary kneader in which mixing blades performs a planetary motion, wherein FIG. 2 (a) is a top view of such planetary kneader, and FIG. 2 (b) is a side view of such planetary kneader. A kneading tank 10 of the kneader shown in this figure is provided with a mixing blade 11a which rotates around a subsidiary spindle a in a direction indicated by an arrow, a mixing blade 11b which rotates around a subsidiary spindle b in a direction indicated by an arrow, and a spindle c which revolves these 2 subsidiary spindles a and b in a direction indicated by an arrow. That is, it is so constructed that the mixing blades 11a and 11b "rotate" around the respective subsidiary spindles a and b, while the subsidiary spindles a and b "revolve" around the spindle c.

As the mixing blades 11a and 11b provided as described above move with complicated loci, the dispersion slurry within the kneading tank 10 can be uniformly kneaded and the coagula can be completely divided. As a result, a large quantity of the inorganic particles can be efficiently dispersed in a comparatively small quantity of aqueous medium.

In FIG. 2, 2 subsidiary spindles a and b are shown. However, it is acceptable that single subsidiary spindle is provided or 3 or more subsidiary spindles are provided instead of 2 subsidiary spindles. Furthermore, when a plurality of subsidiary spindles are provided, each subsidiary spindle may be positioned at regular intervals or at irregular intervals.

Also in FIG. 2, 2 mixing blades are provided as a pair on each subsidiary spindle. However, it is acceptable that single mixing blade is provided on each subsidiary spindle, or 3 or more mixing blades are provided as a pair on each subsidiary spindle.

It is also acceptable that a high-speed blade is provided on a spindle coaxially with or differently from the subsidiary spindle of the mixing blades to improve the ability of dividing and dispersing the inorganic particles.

In FIG. 2, both the spindle c and the subsidiary spindles a and b rotate counterclockwise viewed from the top. However, it is acceptable that the spindle c and the subsidiary spindles a and b are set differently from each other in the rotational direction to have different loci of the mixing blade motion.

In FIG. 2, the mixing blades 11a and 11b are curved and twisted between the ends (twisted shape). However, it is accepted that the mixing blades 11a and 11b are differently shaped in any way as long as such shape ensures that the dispersion slurry within the kneading tank can be uniformly kneaded and the coagula can be completely divided and as a result a large quantity of the inorganic particles can be efficiently dispersed in a comparatively small quantity of an aqueous medium.

Among those planetary kneaders which satisfy the requirements described above, in addition to those kneaders named above, are Universal Mixer/Stirrer ("Universal Mixer/Stirrer" is a tread mark) of Dalton Co., Ltd., Universal Mixer ("Universal Mixer" is a tread mark) of Powlex Co., Ltd., Planetary Kneader/Mixer ("Planetary Kneader/Mixer" is a tread mark) of Ashizawa Co., Ltd., T.K. Hibis Disper Mix ("T.K. Hibis Disper Mix" is a tread mark) of Tokushu Kika Kogyo Co., Ltd., and Planetary Disper ("Planetary Disper" is a tread mark) of Asada Iron Works Co., Ltd.

Preferably, the equipment used in the preliminary dispersion process of the production method (A) or in the production method (B) should be provided with a lining of resin, such as polyurethane, Teflon or epoxy resin, to protect the aqueous dispersion slurry from metallic contamination after production. Still preferably, the liquid contacting portions, such as inner walls and mixing blades, of such equipment should be provided with a lining of ceramic, such as zirconia, to prevent metallic contamination and increase wear resistance.

The production method (B) may be performed for plural times on single equipment or one or more times on a combination of plural, different equipment.

# 3. ADDITION OF ACID OR ALKALI

In the production method (A), an acid or alkali should preferably be added at least to the aqueous medium before the preliminary dispersion process, to the dispersion slurry during the preliminary dispersion process, to the dispersion slurry after the preliminary dispersion process, to the dispersion slurry during the main dispersion process, to the dispersion slurry during the main dispersion process, or to the dispersion slurry after the main dispersion process to adjust the pH of the aqueous dispersion slurry to be finally in a range of 2 to 12. As a result of this addition, the dispersion stability of the resultant aqueous dispersion slurry becomes much higher.



In the production method (B), an acid or an alkali should preferably be added at least to the aqueous medium before the dispersion process, to the dispersion slurry during the dispersion process, or to the dispersion slurry after the dispersion process to adjust the pH of the aqueous dispersion slurry to be finally in a range of 2 to 12. As a result of this addition, the dispersion stability of the resultant aqueous dispersion slurry becomes much higher.

The pH of the aqueous dispersion varies depending on the intended use. However, such pH should preferably be in a range of 3 to 11. If the pH is set to be less than 2 or more than 12, the inorganic particles tends to be dissolved or coagulated due to insufficient colloidal stability.

The acid or the alkali may be added in any one of the stages described above or in any 2 or 3 of such stages. In whatever stage the acid or the alkali is added, preferably, the aqueous medium or dispersion slurry should be continuously stirred while the acid or alkali is added. If the acid or the alkali is added after the dispersion, a part of the inorganic particles of the dispersion slurry may be coagulated due to a shock of localized increase in concentration. To prevent this, it is preferable that the dispersion process should be performed again or high-speed mixing processing should be performed by using a homo-mixer, a disper or the like after the addition of the acid or the alkali.

As acids, inorganic acids, such as hydrochloric acid, nitric acid, sulfuric acid and phosphoric acid, or organic acids, such as acetic acid, phthalic acid, acrylic acid, methacrylic acid, crotonic acid, polyacrylic acid, maleic acid and sorbic acid, may be used. Among of these, hydrochloric acid, nitric acid and acetic acid, which are univalent acids, are preferable. As alkalis, inorganic bases, such as potassium hydroxide, sodium hydroxide, lithium hydroxide and ammonia, and amines, such as ethylenediamine, trirthylamine and piperazine, may be used.

# 4. PREFERABLE RANGE OF AQUEOUS DISPER-SION SLURRY

The average diameter of the inorganic particles of the aqueous dispersion slurry according to the present invention is in a range of 0.01 to 2 µm, should preferably be in a range of 0.02 to 1 µm, and should more preferably be in a range of 0.03 to 0.8 µm.

If such diameter is smaller than 0.01  $\mu$ m, the viscosity of the aqueous dispersion slurry of the inorganic particles will be excessively high, and good dispersion stability can not be obtained. If such diameter is larger than 2  $\mu$ m, the dispersion stability will be so low as to cause sedimentation.

This particle diameter can be controlled by setting a suitable condition in the main dispersion process and/or by selecting a suitable raw material of the inorganic particles. When the high-pressure homogenizer is used,

the particle diameter can be controlled by the pressure, number of processing times, etc. of the high-pressure homogenizer. When the planetary kneader is used, the particle diameter can be controlled by the number of rotations of the mixing blades, the number of revolutions of the subsidiary spindles, the mixing time, etc.

The inorganic particles dispersed in the aqueous dispersion slurry obtained by either of the production methods (A) and (B) according to the present invention are composed of primary particles and secondary particles or of secondary particles alone. The average diameter of the inorganic particles can be measured by using Laser Particle Diameter Analysis System Model LPA-3000S/3100, which is controlled based on a dynamic light scattering method, of Otsuka Denshi Co., Ltd.

#### SPECIFIC DESCRIPTION

Now, the present invention will be specifically described by referring to examples and reference examples. However, the present invention is not limited to such examples.

In the following description, "part" means "part by weight," and "%" means "percent by weight or wt%." The average particle diameter of the inorganic particles in the aqueous dispersion slurry of the inorganic particles were measured by using Laser Particle Diameter Analysis System Model LPA-3000S/3100 of Otsuka Denshi Co., Ltd.

# 1. PRODUCTION METHOD (A)

# [EXAMPLE 1]

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Furned silica (Aerogil #50 of Nippon Aerosil Co., Ltd.) of 15 kg was preliminarily dispersed in 60 kg of ion exchange water while being sucked by using a powder introducer/ mixer/disperser (e.g., Jet Stream Mixer TDS "Jet Stream Mixer TDS" is a tread mark) of Mitamura Riken Kogyo Inc.).

Then, thus obtained preliminarily dispersed slurry were subjected to the main dispersion processing by using a high-pressure homogenizer provided with a sintered diamond dispersion unit (Microfluidizer ("Microfluidizer" is a tread mark) of Mizuho industrial Co., Ltd.) under a pressure of 800 kg/cm<sup>2</sup>.

Then, thus obtained dispersion slurry out of the main dispersion processing were added with potassium hydroxide solution of 20% in concentration while being stirred, so that the pH thereof was adjusted to 10.

Then, under the same conditions as those of the main dispersion processing, the processing by using the high-pressure homogenizer was performed again.

The average particle diameter of thus obtained particles in the aqueous dispersion slurry of the fumed silica was 0.25  $\mu$ m. The aqueous dispersion slurry were left for 30 days at 25°C, but none of increase in viscosity, gelation or sediment was recognized.

# [EXAMPLE 2]

Fumed alumina (Al<sub>2</sub>O<sub>3</sub>-C of Nippon Aerosil Co., Ltd.) of 15 kg and 1.8 kg of 1N nitric acid was preliminarily dispersed in 48.2 kg of ion exchange water while being sucked by using the Jet Stream Mixer TDS ("Jet Stream Mixer TDS" is a tread mark) of Mitamura Riken Kogyo Inc. described above.

Then, thus obtained preliminarily dispersed slurry were subjected to the main dispersion processing by using a high-pressure homogenizer provided with a single crystal diamond dispersion unit (Ultimaizer ("Ultimaizer" is a tread mark) Model HJP-30030 of Sugino Machine Limited) under a pressure of 1,500 kg/cm<sup>2</sup>.

The pH and average particle diameter of thus obtained particles in the aqueous dispersion slurry were 4.1 and 0.16  $\mu$ m, respectively. The aqueous dispersion slurry were left for 30 days at 25°C, but none of increase in viscosity, gelation or sediment was recognized.

# [EXAMPLE 3]

Potassium hydroxide solution in which 60 g of potassium hydroxide particles were dissolved in 9 kg of ion exchange water was preliminarily dispersed while being stirred by using a kneader (T.K. Hibis Disper Mix ("T.K. Hibis Disper Mix" is a tread mark) Model HDM-3D-20 of Tokushu Kika Kogyo Co., Ltd.) and continuously adding 5 kg of fumed silica (Aerogil #90 of Nippon Aerosil Co., Ltd.) for 3 hours.

Then, thus obtained preliminarily dispersed slurry were further kneaded for 2 hours, and then added with ion exchange water to dilute the solid concentration to 30%.

Then, thus obtained preliminarily dispersed slurry were subjected to the main dispersion processing by using a high-pressure homogenizer provided with a single crystal diamond dispersion unit (Genus PY Model PRO2-15 of Genus Co., Ltd.) under a pressure of 500 kg/cm<sup>2</sup>.

The pH and average particle diameter of thus obtained aqueous dispersion slurry of fumed silica were 10 and 0.19  $\mu$ m, respectively. The aqueous dispersion slurry were left for 30 days at 25°C, but none of increase in viscosity, gelation or sediment was recognized.

#### [EXAMPLE 4]

Aqueous dispersion slurry of alumina were obtained by the same method as the example 2 except that instead of using the fumed alumina, metallic vapor deposited alumina (NanoTek ("NanoTek" is a tread mark) of Nanophase Technologies Corporation represented by C.I. Kasei Co., Ltd.) was used.

The pH and average particle diameter of thus obtained aqueous dispersion slurry of alumina were 4.2 and 0.13 µm, respectively. The aqueous dispersion slurry were left for 30 days at 25°C, but gelation or sed-

iment was not recognized.

# [REFERENCE EXAMPLE 1]

Aqueous dispersion slurry of the furned silica was formulated by the same method as the example 1 except that the main dispersion processing was not performed in the example 1.

Thus obtained aqueous dispersion slurry were left, and after 30 minutes, all the quantity of the aqueous dispersion slurry gelled and completely lost fluidity.

#### 2. PRODUCTION METHOD (B)

# [EXAMPLE 5]

Furned aluminum oxide C (Degusa Co., Ltd.) of 10 kg was continuously added for 1 hour to 8.6 kg of ion exchange water in which 1.4 kg of 1N nitric acid had been dissolved while the mixture was kneaded by a planetary kneader having liquid contacting portions of the stirrer and container coated with urethane resin (Universal Kneader ("Universal Kneader" is a tread mark) Model 30DM of Dalton Co., Ltd.) with the subsidiary spindles and spindle of the stirrer in rotation at 60 rpm and 20 rpm, respectively. After such addition, the mixture was further kneaded for another 1 hour at 50% solid concentration.

Thus obtained slurry was further dispersed for another 1 hour at 2,000 rpm by using a non-planetary disperser (T.K. Homodisper ("T.K. Homodisper" is a tread mark) of Tokushu Kika Kogyo Co., Ltd.). Thus obtained slurry was diluted with ion exchange water, and aqueous dispersion slurry of aluminum oxide with 30% solid concentration were obtained. Thus obtained aqueous dispersion slurry were subjected to metallic analysis by atomic absorption spectroscopy. As a result, there was a little metallic contamination with 0.5 ppm of iron.

The pH and average particle diameter of the aqueous dispersion slurry of fumed aluminum oxide were 4.3 and 0.12  $\mu$ m, respectively. The aqueous dispersion slurry were left for 30 days at 25°C, but none of increase in viscosity, gelation or sediment was recognized.

# [EXAMPLE 6]

Furned titanium dioxide (P25, Nippon Aerosil Co., Ltd.) of 2 kg was continuously added for 1 hour to 2 kg of distilled water in which 100 g of acetic acid had been dissolved while the mixture was kneaded by a planetary kneader having liquid contacting portions of the stirrers and container coated with urethane resin (Universal Kneader ("Universal Kneader" is a tread mark) Model 5DM of Dalton Co., Ltd.) with the subsidiary spindles and spindle of the stirrer in rotation at 90 rpm and 30 rpm, respectively. After such addition, the mixture was further kneaded for another 1 hour at 50% solid concen-

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tration.

Thus obtained slurry was diluted with ion exchange water, and aqueous dispersion slurry of titanium dioxide with 40% solid concentration were obtained.

The pH and average particle diameter of thus obtained aqueous dispersion slurry of titanium dioxide were 6.5 and 0.16 µm, respectively. The aqueous dispersion slurry were left for 30 days at 25°C, but none of increase in viscosity, gelation or sediment was recognized.

# [EXAMPLE 7]

Furned silicon oxide (Aerosil #50 ("Aerosil #50" is a tread mark) of Nippon Aerosil Co., Ltd.) of 6 kg was continuously added for 30 minutes to 8kg of distilled water in which 60g of potassium hydroxide granules had been dissolved while the mixture was kneaded by using a planetary kneader having liquid contacting portions of the stirrers and container coated with urethane resin (T.K. Hibis Disper Mix ("T.K. Hibis Disper Mix" is a tread mark) Model HDM-3D-20 of Tokushu Kika Kogyo Co., Ltd.) with the twisted blades rotated around the respective subsidiary spindles and the subsidiary spindle revolved around the spindle at 10 rpm and 30 rpm, respectively.

After such addition, the mixture was further subjected to kneading for another 1 hour at 43% solid concentration while the twisted blades were rotated around the respective subsidiary spindles at 30 rpm and at the same time to disper procesing with the corres type high-speed rotary blades of 80 mm in diameter rotated around the respective subsidiary spindles at 2,000 rpm and the spindle rotated at 10 rpm for both the kneading and disper processing.

Thus obtained slurry was diluted with ion exchange water, and aqueous dispersion slurry of silicon oxide with 30% solid concentration were obtained.

The pH and average particle diameter of the aqueous dispersion slurry of silicon oxide were 10.5 and 0.23  $\mu$ m, respectively. The aqueous dispersion slurry were left for 30 days at 25°C, but none of increase in viscosity, gelation or sediment were recognized.

# [EXAMPLE 8]

Aqueous dispersion slurry of silicon oxide were obtained by the same method as the example 7 except that fumed silicon oxide was added to 8 kg of distilled water in which 60 g of potassium hydroxide granules had not been dissolved and then the mixture was subjected to further kneading and disper processing for another 1 hour by using blades and potassium hydroxide was added as 10% solution at 10 minutes before ending such additional processing.

The pH and average particle diameter of the aqueous dispersion slurry of silicon oxide were 10.3 and 0.21  $\mu$ m, respectively. The aqueous dispersion slurry were

left for 30 days at 25°C, but none of increase in viscosity, gelation or sediment were recognized.

# [REFERENCE EXAMPLE 2](COMPARISON TO EXAMPLE 7)

Aqueous dispersion slurry were formulated by the same method as the example 7 except that a disperser with the stirrers only in rotation (T.K. Homo Disper ("T.K. Homo Disper" is a tread mark) of Tokushu Kika Kogyo Co., Ltd.) was used as a kneader instead of T.K. Hibis Disper Mix of Tokushu Kika Kogyo Co., Ltd. with the stirrers in both rotation and revolution (planetary mechanism).

Complete dispersion slurry could not be obtained. The pH and average particle diameter of the aqueous dispersion slurry were 10.6 and 2 µm or more, respectively. The aqueous dispersion slurry were left for 2 days at 25°C, and gelled and lost fluidity.

# [REFERENCE EXAMPLE 3](COMPARISON TO EXAMPLE 6)

Aqueous dispersion slurry were formulated by the same method as the example 6 except that 6 kg of distilled water in which 100 g acetic acid had been dissolved, instead of 2 kg of distilled water in which 100 g acetic acid had been dissolved, to adjust the solid concentration during kneading to 25%.

Complete dispersion slurry could not be obtained. The pH and average particle diameter of the aqueous dispersion slurry were 6.7 and 2 µm or more, respectively. The aqueous dispersion slurry were left for 2 days at 25°C, and gelled and lost fluidity.

# [REFERENCE EXAMPLE 4](COMPARISON TO EXAMPLE 6)

Aqueous dispersion slurry were formulated by the same method as the example 6 except that 0.6 kg of distilled water in which 100g acetic acid had been dissolved, instead of using 2 kg of distilled water in which 100 g acetic acid had been dissolved, to adjust the solid concentration during kneading to 74%.

During continuous addition of the inorganic particles, the mixer was stopped due to overload, and further continuous kneading operation was impossible.

### Claims

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 A production method for aqueous dispersion slurry of inorganic particles, wherein inorganic particles are dispersed in an aqueous medium to an average particle diameter of 0.01 to 2 μm by using either of a method (A) having a preliminary dispersion process in which said inorganic particles are added to and dispersed in an aqueous medium and a main dispersion process in which dispersed slurry out of

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may be positioned above each hollow cavity as the rounded shoulder access member is rotated.

FIG. 11 shows a side view of a divider 70 while FIG. 12 shows a back view of a divider 70. Each divider includes an innermost edge 74 that interacts with the inner container 25. The divider 70 includes a bottom edge 78 and an outermost curved edge 80. The outermost curved edge 80 is curved to match the tapered portion 61 at the top of the outer container near the upper region 60. The outermost curved edge 80 is in contact with the inner surface 36 of the outer container 30 when the divider 70 is in position within the hollow region 34. Now referring to FIG. 4, the innermost edge 74 is preferably received by a groove 76 on the exterior surface 35 of the inner container 25. Each groove 76 is sized for receiving the divider 70.

In a preferred embodiment, the decorative container 15 includes eight removable dividers. The number of dividers may be varied. For example, six dividers may be used. Alternatively, four dividers may be used. Other numbers of dividers may also be used. Preferably, the dividers are equally spaced throughout the hollow region 34.

Each divider 70 includes a thicker portion 84. As shown in FIGS. 3 and 10, the grooves 76 that receive the dividers 70 are slightly wider at a bottom portion and become narrower at a top portion of the groove 76 in one embodiment. Where the container base 32 is formed using injection molding processes, it is desirable for the divider grooves 76 to include a wider bottom portion to facilitate removal of the molded container base from the mold. A thicker portion 84 of the dividers is provided, adjacent to the bottom edge 78 and adjacent to the innermost edge 74, to occupy the space in the divider grooves so that the dividers 70 are not able to move freely side-to-side within the grooves. The thicker portion 84 of the dividers extends along the length of the divider for about 2 inches or about 5 centimeters in the illustrated embodiment. The thicker portion of the divider is about 5 millimeters, or about .21 inches in thickness. The remainder of the divider has a wall thickness of about .09 inch, or about 2 millimeters. In a preferred embodiment, the width of the dividers is about .871 inches, or about 2.2 centimeters. The preferred length of the dividers is about 7.6 inches, or about 19 centimeters.

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FIGS. 1-4 also illustrate the decorative lid of the present invention. The lid 20 includes a hollow lid region 100 where decorative objects may be displayed. The lid 20 includes a base portion 110 and a top shell portion 115 that are joined together at an outer edge 118 of the base portion 110. The base portion 110 defines a lid access opening 120 for providing access to the hollow lid region 100.

The lid 20 may also include a removable lid member 125 for removably fitting into the lid access opening 120. The removable lid member 125 is shaped to fit into place in the lid access opening 120 and may include raised portions 130 to allow for easier gripping by a user. The removable lid member 125 may fit into the lid access opening 120 using a variety of techniques known in the art, such as a screw on configuration, a snap on configuration, or preferably, a press-fit configuration.

Preferably, the removable lid member 125 is made of a material that is somewhat more flexible than the material that forms the lid base portion 110. The lid access opening 120 may have an edge surface that is angled to interact with a corresponding angled surface on the outer edge of the removable lid member. The angled surfaces and the relative flexibility of the removable lid member, along with the dimensions of the removable lid member 125 and base 110, facilitate a press-fit interaction between the removable lid member 125 and the lid base portion 110. It is preferable that the lid access member can be removed from the lid access opening by hand without any special tools.

The base portion 110 of the lid 20 is shaped to fit into the upper opening of the decorative container 15 defined by the neck 62. The base portion 110 may include a rim 134 that fits inside the neck 62 of the inner container 25 of the decorative container 15.

The lid 20 is also intended to display the contents of the hollow lid region 100. Accordingly, it is preferred that at least a portion of the lid 20 is at least partially transparent. For example, it is preferred that at least the shell 115 of the lid 20 is at least partially transparent. More preferably, all components of the lid are at least partially transparent.

The shell 115 of the lid 20 may be shaped in many different configurations. In the illustrated embodiment, the shell is dome shaped, but many other shapes are possible. The shell 115 may attach to the base portion 110 of the lid using many different configurations. In a preferred embodiment, an outer edge 118 of the base 110 is sized to tightly fit within the

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lower edge 119 of the shell 115. The lower edge 119 of the shell portion may have an angled surface that facilitates this connection. Alternatively, adhesives, thermal or sonic welding or other techniques may be used to join the shell 115 and the base 110 of the lid 20.

The decorative container system 10 of the present invention may be used to store items such as flour, cookies, or cooking ingredients, among other household items, in the interior space 64. The hollow region 34 is intended to serve a decorative function by providing a location to display decorative items such as dried pasta, colorful candies, beads, or photos, for example.

It may be desirable to combine two and three dimensional objects when filling the hollow region of the decorative container. For example, where some of the dividers in the preferred embodiment having eight dividers are removed, a photograph could be placed within a wider hollow cavity. Decorative objects that compliment the photo could be placed in the other hollow cavities. It may be desirable to insert tissue paper or other filler material between the back of the photograph and the inner container, so that the photograph is pressed against the outer container. Many different arrangements of decorative objects may be accomplished because of the flexibility provided by the removable dividers in one embodiment of the invention.

In order to serve the decorative purpose, it is preferred that the outer container 30 is at least partially transparent. By "at least partially transparent" it is meant that an ordinary observer can discern the objects within the hollow region 34. More preferably, all components of the decorative container system are at least partially transparent. In a preferred embodiment, some or all of the components are clear.

It is also possible that some portions of the decorative container system are transparent, while others are opaque. For example, the inner container 25 may be opaque, while the outer container, dividers, and other components may be transparent. In the alternative, the inner container, dividers and bottom plate may be opaque, while only the outer container is transparent.

The decorative container 15 may be provided in many different sizes, depending on the types of decorative items to be displayed and the types of objects to be stored within the container. In one preferred embodiment, the container is cylindrically shaped and the upper rim of the container is circular. Preferably, the upper rim of the container has a diameter of about 3 to 8 inches, or about 8 to 20 centimeters, preferably about 5 inches, or about 13 centimeters. In the preferred embodiment, the outer diameter of the container is about 5 to 10 inches, or about 13 to 25 centimeters and is preferably about 7 inches, or about 18 centimeters. The height of the container in the preferred embodiment may be about 4 to 12 inches, or about 10 to 30 centimeters, preferably about 8 inches, or about 21 centimeters. In the preferred embodiment, the distance between the inner container and the outer container may be about .5 inch to 2 inches, or about 1.25 to 5 centimeters, preferably about 1 inch or about 2.5 centimeters.

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... 20 The preferred embodiment is cylindrically shaped, which allows for the dividers to be easily removed through the access opening. Other shapes are also possible according to the present invention, such as bowl shapes or tray shapes. The walls of the decorative container system are preferably about 0.09 inches, or about 2.3 millimeters thick.

The decorative container system may be constructed of many different materials that have adequate rigidity for storing items in the open area and for storing decorative objects in the internal cavities. Other desirable characteristics of the decorative container are transparency, ease of manufacture, low cost, durability, washability, and visual attractiveness. Preferably, the components of the decorative container are made of the same material and are molded using well known techniques. Preferred materials for the decorative container include polystyrene and other plastic materials. In a preferred embodiment, the components of the decorative container system are all made of polystyrene except for the removable lid access member which is made of polypropylene.

Many different construction techniques may be used to produce the decorative container system of the present invention. Preferably, injection molding is used to produce the components of the decorative container. In one embodiment, each of the components that is separately pictured in the exploded view FIG. 4 is produced as a separate component using injection molding. The container base 32 is molded as one piece, including the inner and outer containers and the neck. The injection molding process may result in small lines on the surface of the components called "witness lines" where two mold pieces meet or where there is a change in the radius of curvature of a surface. In FIG. 1, witness lines 140 are shown on

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the shell portion 115 of the lid and on the outer container wall where there is a change in the radius of curvature. The witness lines will be nearly invisible when the proper molding techniques, known in the art, are employed. Where components of the decorative container system may be permanently attached together, such as the attachment between the shell and the base portion of the lid, adhesive or other well known joinery techniques may be used.

Alternatively, the inner container and the outer container may be separate pieces. The inner container may be removable from the outer container by the user.

The decorative container system of the present invention allows for custom design of a decorative container so that the decorations may be suited for a certain room's décor, the theme of a certain season or for a personal gift with favorite objects of the gift receiver. The removable access member provided for the lid and for the container allow for the contents to be easily changed.

In order to decorate the decorative container system of the present invention, the container 15 is turned upside down and the removable access plate 40 is removed, which provides access to the hollow region 34, as shown in FIG. 13. It may be desirable to rest the upper neck 62 on a working surface while decorating the container. The dividers 70 may be positioned within the grooves 76 as desired. Then, decorative objects 150 are dropped into position in the various internal cavities. A wand or other positioning member may be used to assist with placing the decorative objects within the cavities. An example of a wand that may be used with the decorative container of the present invention is described in U.S. Patent Application Serial No. 09/586,153, which is hereby incorporated herein by reference in its entirety, in the text related to FIGS. 7-9. When the decorative objects have been arranged as desired, the removable access member is put back in place in the access opening. Once the removable access member is in place, the decorative objects are securely contained within the internal cavities and the container 15 may be turned so that the opening to the interior space is facing upward.

The lid 20 of the system 10 may be decorated by first removing the removable lid member 125. Decorative objects may then be placed within the hollow lid region 100. Then the removable lid member 125 is pressed into place in the lid access opening 120. The lid 20 may be placed in position on top of the container 15.

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When a change of decorative items within the internal cavities is desired, the removable access member may be removed and the objects may be emptied from the internal cavities. Likewise, the removable lid member 125 may be removed from the lid access opening 120 and the objects may be emptied from within the hollow lid region 100.

An open area 160 is present at the bottom of the container base 32 as shown in FIG. 13. In the preferred embodiment, the access member 40 will occupy most of the open area 160, as shown in the cross-sectional view of FIG. 13. However, in an alternative embodiment, the open area 160 and access member 40 would be configured to allow objects to be placed within the open area.

The various embodiments described above are provided by way of illustration only and should not be construed to limit the invention. Those skilled in the art will readily recognize various modifications and changes which may be made to the present invention without strictly following the preferred embodiments and applications illustrated and described herein, and without departing from the true spirit and scope of the present invention which is set forth in the following claims.